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A USER'S GUIDE TO THE PICKER DIFFRACTOMETER FOR POLYMER MORPHOLOGY STUDIES



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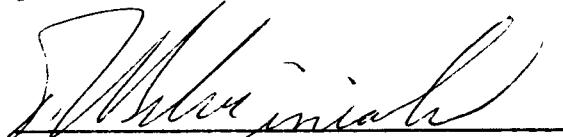
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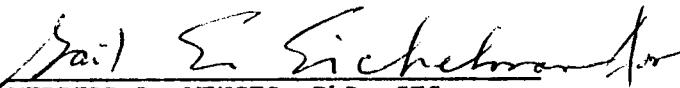


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FOREWORD

This report was prepared by the Polymer Branch, Nonmetallic Materials Division, and Vanderbilt University (through Universal Energy Systems, Inc.) under contract F33615-82-C-5001 to the Materials Laboratory. The work was initiated under Project 2302, "Research to Define the Structure Property Relationships," Task No. 2303Q3, Work Unit Directive 2303Q307, "Structural Resins." Dr Thaddeus E. Helminiak served as the AFWAL/ML Work Unit Scientist. Co-authors were Dr P. Galen Lenhert, Vanderbilt University, and Lt Joseph F. O'Brien and Dr W. Wade Adams, Materials Laboratory (AFWAL/MLBP). This report covers research conducted from 1980-1984.

TABLE OF CONTENTS

SECTION		PAGE
I	INTRODUCTION	1
II	GENERAL COMMENTS ON THE PICKER FACS-I SYSTEM	2
III	CHOICE OF INSTRUMENTAL PARAMETERS	4
IV	CRYSTALLITE SIZE DETERMINATION IN POLYMER SAMPLES	6
V	ORIENTATION FACTOR MEASUREMENTS: RESOLVED PEAKS	23
VI	DETERMINATION OF ORIENTATION FOR POLYMER FIBERS: OVERLAPPING REFLECTIONS	28
VII	DIFFRACTOMETER SCANS ALONG LAYER LINES FOR POLYMER MATERIALS	30
VIII	POLE FIGURE DATA COLLECTION AND ANALYSIS	34
	REFERENCES	41

LIST OF FIGURES

FIGURE NO.		PAGE
1	HMTA in 0.5 mm Capillary as Crystallized.	7
2	HMTA Recrystallized and Ground.	
	(a) 0.27 mm Capillary Exposed 8 hours	8
	(b) 0.5 mm Capillary Exposed 1 Hour	8
3	Plot of the HMTA Data in Table 1.	10
4	Graph of ABPBI-PBT Blend Equatorial Scan.	19
5	Plots of Full Width at Half Maximum (in Units of $\frac{2\sin\theta}{\lambda}$) Standards.	
	(a) Equatorial Geometry, Sample Diameter vs Δs for Reflections at 17.85° and 31.20° 2θ .	20
	(b) Meridional Geometry, s vs Δs , Data for 0.29 mm and 0.54 mm Capillaries.	21
6	Plot of First Layer Line for ABPBT Fibers.	33
7	Pole Figure Geometry, Reflection and Transmission Methods.	34

LIST OF TABLES

TABLE NO.	PAGE
1 TWO-THETA SCAN OF MMTA FACS-I INPUT AND OUTPUT	9
2 PEAK FIT OF THE DATA IN TABLE 1	12
3 TWO-THETA SCAN OF HEAT-TREATED ABPBI-PBT FIBERS	15
4 EXAMPLE PRINTOUT CHECK FOR TAIL OF A PBO DISTRIBUTION	24
5 EXAMPLE ORIENTATION FACTOR RUN FOR PBO (AS-SPUN) #042083N1	25
6 ORIENTATION FACTORS FOR TWO PBT REFLECTIONS	26
7 ORIENTATION FACTORS FOR VARIOUS SVA HEIGHTS	26
8 PRECISION OF ORIENTATION FACTOR	27
9 EXAMPLE OF FACS-I INPUT-OUTPUT FOR A LAYER LINE SCAN FOR A HEAT-TREATED ABPBT SAMPLE	32
10 EXAMPLE OF TAPE HEADING	35
11 TRANSMISSION MODE POLE FIGURE INPUT AND OUTPUT	36
12 TWO-THETA SCAN FOR A BUNDLE OF PBT FIBERS	37
13 UNIAXIAL SCANS FOR PBT	38
14 UNIAXIAL SCAN FOR PBT WITH BACKGROUNDS MEASURED FOR 20 S EACH, 1.3° ABOVE AND BELOW THE PEAK POSITION	38

SECTION I

INTRODUCTION

The Picker FACS-I automated x-ray diffractometer has been an integral part of the Polymer Branch Morphology Laboratory since 1975, when it was moved from the Metals Division to its present home in the Nonmetallic Division in Building 56. Although designed originally for use solely as a single-crystal diffractometer, it has been used extensively in the Polymer Branch Morphology Laboratory for polymer analysis. Modifications to the control software on the PDP 8/I computer have enabled the scientist to collect data on semi-crystalline or amorphous polymer specimens, in order to study crystallite orientation and crystallite size, shapes of amorphous halos, and intensity distributions for polymer structure analysis.

This report provides essential information for use of the Picker system for polymer analysis. It is not intended to be all inclusive; as more experiments are performed, modification will be necessary. In addition, as the equipment is updated, the manual will be revised accordingly.

SECTION II

GENERAL COMMENTS ON THE PICKER FACS-I SYSTEM

The FACS-I automated diffractometer was designed to collect x-ray diffraction data for single-crystal structure analysis. It can also be used to make diffraction measurements on polycrystalline materials for orientation studies, crystallite size determination or for two-dimensional pole figure analysis. In general the instrument parameters are similar for both types of experiments. The sample, sample holder and measurement procedures differ.

The instrument consists of a PDP 8/I computer which is interfaced to the goniostat (diffractometer), the x-ray tube shutter and the diffracted beam filter wheels. The computer controls each of the four goniostat angles (2θ , ω , χ , and ϕ) by means of a motor used to drive the angle and an encoder which reads the angle positions to 0.01° . The control program accepts commands which are converted by the computer to appropriate electrical signals and sent to the hardware. Commands are entered into the computer through the terminal keyboard. As discussed below, simple commands allow each hardware function to be activated individually. Other commands cause the computer to carry out complex functions such as the various data collection modes.

The x-ray tube is independent of the computer and operates continuously at voltage and current settings selected manually. The x-ray beam from the tube can be "reflected" from a monochromator crystal, allowed to pass directly to the sample or filtered by an appropriate beta filter. An incident beam collimator allows a narrow beam of x-rays to fall on the sample. X-rays scattered (diffracted) by the sample are detected when they enter the diffracted beam collimator and fall on the detector surface. This collimator serves mainly to keep radiation scattered by the air from entering the detector. The angular resolution of the instrument is determined by the x-ray source size, the area (or volume) of the sample illuminated, and the detector aperture (SVA) which can be varied symmetrically in both vertical and horizontal width.

X-ray quanta that enter the detector produce electrical pulses which are amplified by a preamplifier connected directly to the photomultiplier tube in the detector. These pulses are sent to the pulse height analyzer and, if they pass the energy discrimination criteria which have been set manually, are counted by the scaler. The scaler unit also contains a digital clock (timer). Both the scaler and the timer can be started, stopped and read by the PDP 8/I computer.

The use of the computer and the diffractometer control programs are discussed in the manual for the Vanderbilt Disk-Oriented Diffractometer System (Lenhert, 1974). The manual should be consulted (and studied) before using the computer. The elementary commands allow manipulation of individual angles, shutter, timer, scaler, etc. Other commands are used to carry out scans, execute a series of pole figure measurements, write the results on magnetic, etc.

X-ray tubes designed for diffraction usually have molybdenum or copper targets. The K α line is prominent for both materials. In some experiments the full spectrum of the tube is used, but more often a filter is inserted to partially remove the unwanted portion of the spectrum. If a high degree of monochromaticity is required a monochromator crystal must be used. Documentation supplied with the x-ray tube will show the operational limits of the tube in use. The standard operating voltage for Mo tubes is 50 Kv. Cu tubes are usually run at 30 to 40 Kv. Tube design and focal spot size determine the maximum loading and, therefore, the highest current setting allowed.

SECTION III

CHOICE OF INSTRUMENTAL PARAMETERS

GONIOSTAT ALIGNMENT

Alignment of the x-ray tube and the four-circle goniostat is an exacting task and should be undertaken only by one very experienced in the use of the FACS-I system. Alignment procedure and hints are described by Lenhert (1978). If, after studying the alignment procedure you feel alignment is needed, and believe you are competent to undertake the task, go ahead only after obtaining permission from the person responsible for the diffractometer.

ANALYZER SETTINGS

Before making any x-ray measurements, the analyzer settings should be checked. They should be set to accept 90% to 95% of the counts at the wavelength to be used for the experiment, usually Mo K α or Cu K α . The analyzer should reject all counts which differ from this wavelength by more than 30% to 40%. Rejection is better for the shorter wavelength, i.e., Mo K α .

Analyzer settings which will provide a usable band pass are 100% window, gain at 10, upper rejection level at 7.0, lower rejection level at 4.0. For Mo K α the high voltage (detector voltage) should be set to 5.20 (1 Kv or a little more). The high-voltage settings will need to be adjusted if you change to a different radiation. It must also be checked at least once a month; since the NaI(Th) crystal-photomultiplier detector ages, the setting will have to be adjusted. It should be checked before any major experiment.

The correct high-voltage setting may be determined by obtaining a monochromatic beam from a single crystal or the monochromator. (CAUTION: If you use the monochromator, be sure the SVA is closed far enough to prevent damage to the detector, since the beam stop must be removed and the detector will be subjected to the direct beam from the monochromator.) With the shutter open and the monochromatic beam directed at the detector, check the count rate at various high-voltage settings. Adjust the high voltage for maximum count rate.)

With the analyzer set as described, the normal background and system noise will give about one count per minute (CPM) with x-rays ON and the shutter closed. If you get more than 1 CPM you probably have a hardware problem.

MONOCHROMATOR

To insert the monochromator, remove the monochromator housing cover, slide the monochromator "boat" into place, tighten the screw and replace the cover. Next set the two monochromator angles, 2θ ^{mono} and ψ to the values recorded in the FACS-I Log Book. You should now have a strong uniform monochromatic beam when the shutter is opened. The beam uniformity may be conveniently checked by using a pinhole aperture mounted on a sample holder and placed in the x-ray beam. If the pinhole is offset and the X circle is rotated, the beam intensity can be checked on a circular locus of points. For details see Lenhert (1980).

DIFFRACTOMETER PARAMETERS

The effective source size, collimator size, sample size and shape, and the diffracted beam aperture must be optimized for best results. The choice of these parameters determines the intensity of the diffracted beam and the resolution of the experiment. Increased resolution is usually obtained at the expense of intensity, so a compromise must be reached which will give the resolution required without excessive counting times. Each experiment has its own requirements but general rules are helpful, and in many cases standard settings may be used.

The rule with fewest exceptions applies to collimator size. For general use with both single crystals and polycrystalline materials, use the 1.0 collimators. The incident beam and diffracted beam collimators are not interchangeable! The collimator with circular grooves goes on the diffracted beam side.

The effective x-ray source is adjusted by changing the x-ray tube take-off-angle (TOA). High-resolution experiments require a small source size which is obtained with a small TOA. For cell constant determinations with single crystals one might use a TOA of 1.0°. Single crystal data collection is normally done with TOA settings of 2.5° to 3.5°. Polymer materials usually give more diffuse diffraction patterns and TOA settings of 3.0° to 4.0° are normal settings.

The diffracted beam aperture is determined by the setting of the symmetrically variable aperture (SVA). Small settings are used when high resolution is required. Data for use in calculating single-crystal cell constants may be taken with SVA settings of 1.0mm vertical opening and 1.0mm horizontal width. Single crystal data collection is normally done with an SVA setting of 3.75mm x 3.75mm. Experiments with polycrystalline materials sometimes require resolution only with respect to 2θ . For such experiments the SVA should be set wide open in the vertical direction. If resolution is required (for example with pole figure or orientation measurements) the vertical SVA opening will usually be restricted to a range of 2mm to 4mm. A similar range will usually be satisfactory for horizontal SVA settings when 2θ resolution is important to the experiment.

The optimum sample size is often set by the form of the material available. If the experiment requires that the entire sample must be illuminated by the x-ray beam then the sample must have a maximum dimension no greater than 1.0mm. Fiber samples should have a cross section of 1mm or less but the effective sample size along the fiber bundle will be determined by the dimensions of the beam. Samples in the form of sheets will extend beyond the beam in two directions and will have an effective size determined by the x-ray beam cross section.

In many experiments choice of diffraction parameters will be critical. These cases are discussed in the following sections devoted to the different types of measurements.

SECTION IV

CRYSTALLITE SIZE DETERMINATION IN POLYMER SAMPLES

Bragg diffraction peaks have a finite width due to the finite size of the crystallites giving rise to the diffraction pattern. However, this is only one of several reasons why Bragg reflections are, in practice, not of infinitesimal width. Lattice distortions of various sorts, along with instrumental factors and sample size, all increase the breadth of Bragg peaks for a real sample measured on a diffractometer with finite x-ray source and aperture.

PREPARATION AND USE OF HEXAMINE STANDARDS

The usual method of correcting for instrumental factors is to use a standard substance with large enough crystallite size to remove this cause of line broadening. In our study, hexamethylenetetramine (HMTA) from Aldrich was recrystallized by Mr Al Sicree. HMTA is somewhat hygroscopic and the recrystallized material of July 14, 1982 is stored in a desiccator under vacuum. It should be pumped to restore the vacuum immediately after removing a sample.

The crystallites in the recrystallized material are much too large to give a uniform powder pattern. The photograph, figure 1, made from unground recrystallized material shows this. If unground material is used on the diffractometer to make theta scans the peak contains fine structure due to individual crystallites. The peak shapes are therefore not an accurate reflection of the instrumental and sample size parameters and should not be used to correct polymer diffraction scans for these factors.

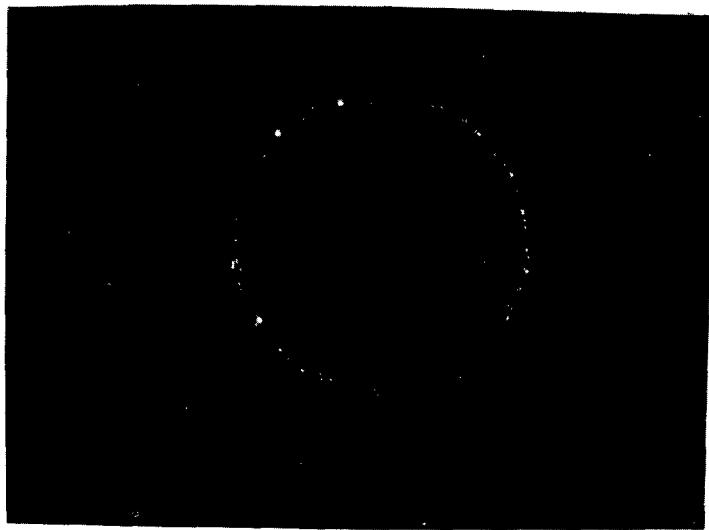
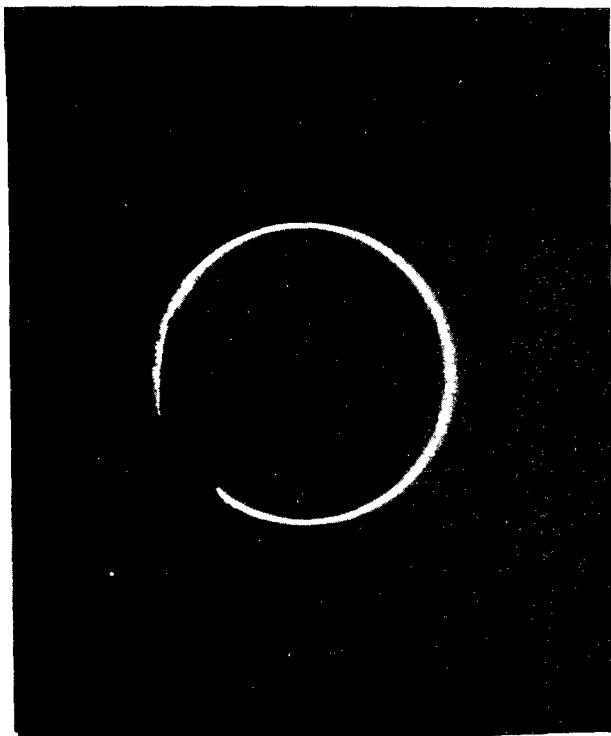


Figure 1. HMTA in 0.5 mm Capillary as Crystallized. Cu K α , Ni filter, 30 Kv, 20 ma.

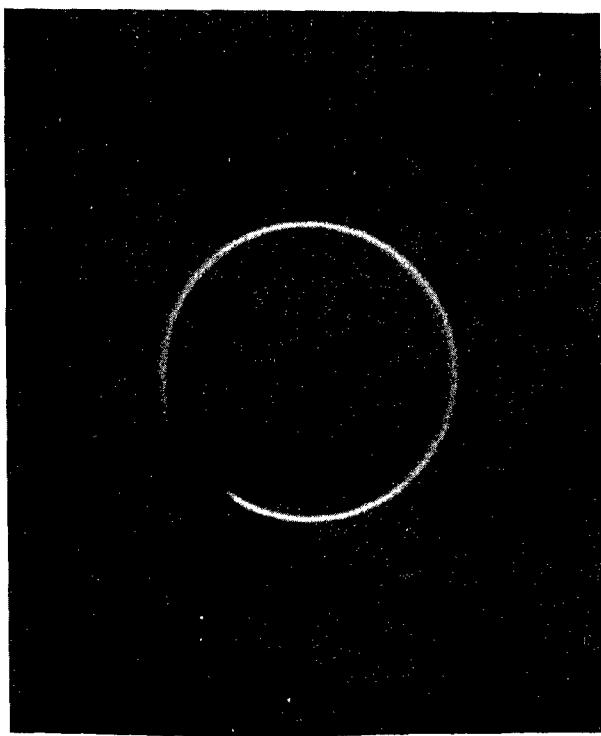
Experience shows that HMTA powder satisfactory for use as a standard can be obtained by grinding the recrystallized material with a mortar and pestle. Be careful not to try to grind too much material at one time. It takes very little to fill a capillary tube. Grind it for 30 minutes or so taking care to work over all parts of the batch you are grinding. Scrape it off the sides of the mortar every few minutes to be sure it is evenly ground.

Fill the capillary tubes by introducing a small amount in the open end and working it to the bottom by stroking the top of the tube with a file to make the tube vibrate and by poking it carefully down the tube with a small tube or a glass fiber. When the bottom 1 to 2 cm of the tube is filled, use the small glass burner and seal the open end. You will find this easy to do if you pull on both ends of the capillary as you put it into the flame. Complete the seal by heating the tip of the sample tube to form a small bead. Mount the sample tube (which should be about 2 to 3 cm long) in one of the small aluminum sample holders. Finally, mount it on a goniometer head.

A photograph taken with the precession camera (no precession motion) should show no more structure than those in figure 2. Samples of 0.27 mm, 0.5 mm and 0.7 mm have been prepared and their powder diagrams measured on the diffractometer.



(a) 0.27 mm capillary exposed 8 hours



(b) 0.5 mm capillary exposed 1 hour

Figure 2. HMTA Recrystallized and Ground. Capillary horizontal in precession camera (no precession motion). Prepared August 2, 1982, Cu K α Ni filter 30 Kv 20 ma.

Sample size is one of the parameters that determines the broadening of a powder line. Therefore, to correct for sample size, one must use a standard sample the same size as the polymer sample being investigated. Consider also that the effective sample size is equal to the actual sample size only if the sample is smaller than the x-ray beam. If the sample is horizontal (as with a meridional scan) the effective sample size in the direction of interest will be determined by the width of the x-ray beam. Therefore a scan at $\chi = 0$ must be made for use with equatorial scans and one at $\chi = 90$ (capillary horizontal) for use with meridional reflections.

The quality of the scans obtained with the standards was greatly improved by grinding the HMTA but continuous rotation of the sample (the phi angle on the FACS-I) improved them further. Phi rotation is obtained by setting the PDP 8/I switch registers to 7776 when the scans are started. Use the /TH command of DIFF, write the data on magnetic tape, enter it into the off-site computer via NFRATINI and plot and analyze it in the usual way. Be sure that you make your scans with fine enough 2θ steps in the region of the peaks you intend to use. Be sure to count long enough to get good counting statistics i.e., 1000 + counts for the peaks and at least 200 - 300 counts for all points on the scan.

The following input (Table 1) was used to obtain satisfactory scans with our samples prepared as described above. Figure 3 shows a plot of the data in table 1.

TABLE 1. TWO-THETA SCAN OF HMTA FACS-I INPUT AND OUTPUT

/DC

90

/PT

8

/FM

/HD

HEXASTD3 .5MM CAPILLARY MERIDIONAL SCAN

/TH

TH=14 DLTH=.5 TH=16 DLTH=.05 TH=19 DLTH=.5 TH=29

DLTH=.05 TH=32.5 DLTH=.5 TH=43 DLTH=.05 TH=46 DLTH=.5 TH=50

DLTH=0

1.90	1.91	2.06	2.10	2.38	2.38	2.31	2.56	2.27	2.45
2.40	2.42	2.36	2.39	2.48	2.29	2.32	2.24	2.38	2.46
2.28	2.59	2.74	2.77	3.09	3.16	3.52	3.91	4.35	7.16
12.49	21.88	32.19	42.74	53.05	63.23	72.10	80.56	91.51	101.47
106.23	107.73	105.65	101.15	90.63	80.59	72.94	62.33	53.11	43.40
31.97	20.57	11.30	6.08	4.64	4.58	4.10	3.97	4.04	4.48
4.15	4.05	4.18	4.38	4.09	3.78	3.15	2.92	2.94	2.74
2.85	3.05	2.74	2.95	3.02	2.79	4.79	5.80	3.00	2.85
2.83	2.81	2.96	2.63	2.81	2.58	2.62	2.78	2.97	2.79
2.82	2.86	2.78	2.63	2.53	2.71	2.74	2.89	2.72	2.87
2.53	2.97	2.80	2.59	2.68	2.62	2.61	2.85	2.82	2.67
2.88	2.81	3.06	2.90	3.32	3.34	3.96	4.86	6.20	7.05
9.05	11.10	13.17	14.50	15.37	16.92	17.67	18.27	17.86	17.81
16.40	14.63	13.03	11.58	10.56	9.05	7.06	5.41	4.11	3.35
3.14	2.84	2.91	2.76	2.88	2.63	2.68	2.83	2.70	2.59
2.61	2.81	2.62	2.76	2.69	2.45	2.48	2.46	2.15	2.19
2.11	4.11	3.32	1.98	1.92	2.04	1.88	1.82	1.96	2.09
2.09	2.07	1.90	1.94	1.93	2.61	2.39	2.45	2.41	2.21
2.40	2.52	2.38	2.35	2.32	2.30	2.28	2.16	2.00	2.10
2.12	2.23	2.18	2.32	2.26	2.26	2.54	3.03	3.28	3.90
4.65	4.85	5.88	6.61	7.27	8.17	9.00	9.91	9.79	9.64
9.69	9.29	8.12	7.90	7.36	6.11	5.45	4.45	3.43	2.81
2.59	2.24	2.19	2.16	2.09	2.18	2.04	1.90	2.01	2.05
2.00	2.13	1.79	2.03	2.05	2.15	2.07	1.75	1.75	3.30
7.12	2.62	1.80	1.92						

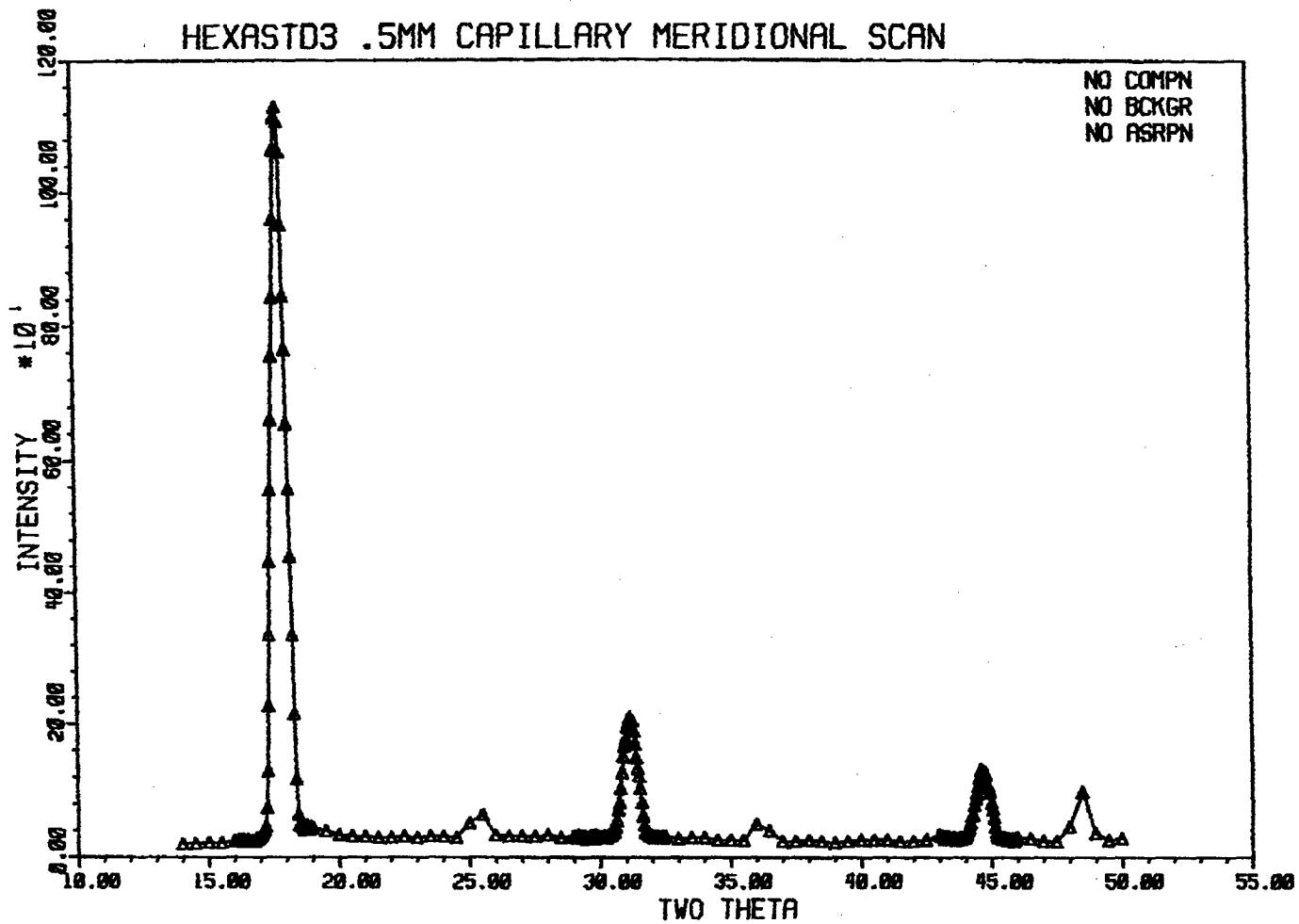


Figure 3. Plot of the HMTA Data in Table 1. Intensity in counts per second.
Cu K α with graphite monochromator.

DETERMINATION OF THE INSTRUMENTAL BROADENING PARAMETER WITH HMTA STANDARDS

Measurement conditions previously used for polymer samples (SVA located 230 mm from the sample, open to 3 x 3 mm and a tube TOA of 3.0°) were found to give diffraction peaks with flat tops with HMTA because the intrinsic width of the HMTA Peaks was smaller than the extended, uniform x-ray source. It was subsequently found that an x-ray tube TOA of 1.0° (1 cm actual focal spot length) which corresponds to an angular width of about 0.04°, and an SVA width of 0.5 mm, which corresponds to an angular width of about 0.12°, gave peaks which could be fit with Gaussian profiles using an available computer program (Anderson, 1985). The following example (table 2) shows the input and output of the peak fit program using the data in table 1. The scan is made with Cu K α monochromatized radiation and a 2θ step size varying from 0.5° in the background region to 0.05° in the peak region. Fits can be obtained with peak step sizes of 0.05° but ease of fit is increased if smaller steps are used in the peak region.

TABLE 2. PEAK FIT OF THE DATA IN TABLE 1. LOW ANGLE PEAK ONLY.

TYPE THE NUMBER OF PEAKS AND BASELINE POINTS
EG 3 (MAXIMUM OF 6 FOR EACH)

1
2

TYPE THE POSITIONS OF THE BASELINE THEN INTENSITIES

1 FIXES THE PARAMETER θ DOES NOT EG:

1.222 2.333 3.444 4.555
 θ 11.222 θ 22.333 1 33.444 1 44.555
.16932 .22525
1 2.0 1 3.0

PEAK POS INT AT MAX HALF-WIDTH GAUSS

1 2.12345 1 30.12345 θ 0.12345 θ 0.12 FOR EXAMPLE

θ .20123 100.0 θ .005 θ .25

YOU HAVE MADE AN ERROR TYPING IN THE FITTING PARAMETERS, RETYPE THE LINE YOU JUST TYPED.

θ .20123 θ 100.0 θ .005 θ .25

* HMTA 0.54MM CAPILLARY MERIDIONAL SCAN *

WEIGHT FACTOR=1/Y(I)

FOR ITERATION > 1 SE = 0.693623E-05
FOR ITERATION > 2 SE = 0.115529E-05
FOR ITERATION > 3 SE = 0.788330E-06
FOR ITERATION > 4 SE = 0.787355E-06
FOR ITERATION > 5 SE = 0.787345E-06

THE AREAS UNDER THE VARIOUS PEAKS ARE GIVEN BELOW:

AREAS = 0.83448

INTEGRAL BREADTH = 0.00724

BASELINE AREA = 0.13984

FINAL FITTING PARAMETERS USED

PEAK POS INT AT MAX HALF-WIDTH GAUSS
 θ 0.20123 θ 115.21251 θ 0.00680 1 1.00

BASELINE POSITIONS AND INTENSITIES

0.1693 1 2.00000

0.2253 1 3.00000

DO YOU WANT A PRINT-OUT OF THE FITTED DATA;

YES

TABLE 2 CONTINUED

TWO THETA	S (1/A)	++++	INTENSITIES	++++	INDIVIDUAL PEAKS	
		OBSERVED	CALCULATED	OBS-CALC	BASELINE	PK 1
15.00	0.1693	2.140	2.000	0.140	2.000	0.000
15.50	0.1749	2.187	2.100	0.087	2.100	0.000
16.00	0.1805	2.485	2.201	0.284	2.201	0.000
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17.55	0.1979	66.597	61.596	5.001	2.511	59.085
17.60	0.1985	75.961	75.046	0.915	2.521	72.525
17.65	0.1990	84.899	88.282	-3.382	2.531	85.751
17.70	0.1996	96.468	100.199	-3.731	2.541	97.658
17.75	0.2001	106.999	109.679	-2.680	2.551	107.128
17.80	0.2007	112.052	115.757	-3.705	2.561	113.196
17.85	0.2012	113.668	117.781	-4.114	2.571	115.211
17.90	0.2018	111.507	115.533	-4.026	2.581	112.952
17.95	0.2024	106.789	109.259	-2.470	2.591	106.669
18.00	0.2029	95.711	99.634	-3.922	2.601	97.033
18.05	0.2035	85.134	87.638	-2.503	2.611	85.027
18.10	0.2040	77.076	74.391	2.685	2.621	71.770
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19.00	0.2141	4.346	2.806	1.540	2.801	0.006
19.50	0.2197	4.030	2.900	1.129	2.900	0.000
20.00	0.2253	3.369	3.000	0.369	3.000	0.000

Similar scans were made for cylindrical samples which measured 0.54 and 0.69 mm in diameter. The reflections at 17.85° and 31.20° were scanned for all samples. The full width at half maximum (FWHM) was obtained by computer fit and plotted versus sample diameter for each reflection. For these samples the FWHM represents the instrumental broadening as a function of sample size because the crystallite size is large. Values for sample sizes in the range from 0.25 mm to 0.70 mm can be read from the curves. Since FWHM is a function of 2θ , one must interpolate for reflections at other diffraction angles.

Equatorial scans of fiber bundles of polymer materials were made and all peaks in the region of interest (0° to 40° 2θ) were fit. The following example, table 3, shows a scan with 4 peaks and the calculation of peak parameters for a similar scan. FWHM in units of $2\sin\theta / \lambda$ are tabulated. The results of the curve fit are shown in figure 4. Crystallite size was calculated from the relations:

$$\Delta\beta_t^2 = \Delta\beta_{\text{obs}}^2 - \Delta\beta_{\text{inst}}^2$$

and

$$L = \frac{K}{\Delta\beta_t},$$

where $\Delta\beta_t$ is the FWHM due to crystallite size, $\Delta\beta_{\text{obs}}$ is the FWHM observed and $\Delta\beta_{\text{inst}}$ is the FWHM due to instrumental factors (as determined from the HMTA scans). L then gives the crystallite size in Å when the constant K (unity in our case) is divided by $\Delta\beta_t$. The results are plotted in figure 5 for both equatorial and meridional geometry.

Note that the effective sample size will change for a given sample when one goes from the equatorial orientation to the meridional orientation where the x-ray beam diameter rather than the sample itself effectively defines the sample size.

TABLE 3. TWO-THETA SCAN OF HEAT-TREATED ABPBI-PBT FIBERS INCLUDING PEAKS AT 11.16, 14.4° AND 26.0° FOLLOWED BY PEAK PARAMETERS FROM LEAST-SQUARES FIT.

/FM

/PT
8

/TH

TH=3	DLTH=.5	TH=8	DLTH=.2	TH=33	DLTH=.5	TH=40	DLTH=0
24.78	15.10	11.01	8.99	7.95	7.39	7.07	7.23
8.83	9.29	10.08	10.20	11.74	12.76	13.84	15.87
23.48	26.69	31.67	36.63	41.73	46.10	51.11	53.66
51.39	47.41	42.75	39.17	35.27	33.63	31.82	30.91
31.00	30.88	31.60	31.42	31.07	30.56	29.76	29.00
24.74	24.06	22.70	21.97	21.18	20.05	20.16	18.81
18.93	19.13	19.22	18.72	19.01	19.43	19.59	19.29
20.57	20.86	21.33	21.80	22.63	23.58	24.52	24.56
28.38	29.05	30.65	32.24	34.16	37.18	39.13	42.33
54.42	62.02	67.23	76.49	84.55	93.95	102.36	112.26
125.43	123.94	117.22	108.28	98.28	87.32	75.09	66.58
42.77	38.30	34.33	29.18	26.14	23.77	21.94	19.75
15.63	13.94	12.93	12.13	11.16	10.51	9.54	9.18
7.59	7.24	6.59	6.46	6.14	6.08	5.18	5.09
4.14	3.92	3.91	3.51	3.65	3.76	3.59	3.58
							3.54

TABLE 3 CONTINUED

DO YOU WANT TO FIT THIS DATA;
YES

WEIGHT FACTOR?
0=SQRT(Y(I)/YMAX)
1=1/Y(I)

1

FROM WHAT INITIAL S TO WHAT FINAL S ;
0.07 1.0

THE MIN S IS AT 0.06789 THIS IS THE 7 DATA POINT
THE MAX S IS AT 0.44366 THIS IS THE 150 DATA POINT

THE TOTAL NUMBER OF DATA POINTS TO BE FITTED IS 144
THE AVERAGE S IS 0.24343 AND THE AVERAGE RCI IS 34.35187

TYPE THE NUMBER OF PEAKS AND BASELINE POINTS
EG 3 (MAXIMUM OF 6 FOR EACH)

4 2

TYPE THE POSITIONS OF THE BASELINE THEN INTENSITIES

1 FIXES THE PARAMETER 0 DOES NOT EG:
1.222 2.333 3.444 4.555
0.11.222 0 22.333 1 33.444 1 44.555
0.06789 0.44366
1 3.0 1 2.0

PEAK POS INT AT MAX HALF-WIDTH GAUSS
1 2.12345 1 30.12345 0 0.12345 0 0.12 FOR EXAMPLE
0 .13 0 45.0 0 .05 0 .5
0 .17 0 20.0 0 .05 0 .5
0 .28 0 30.0 0 .05 0 .5
0 .30 0 50.0 0 .05 0 .5

* ABPBI-PBT HT EQUATORIAL SCAN... CHI=0.0 DEGREES CU T *

WEIGHT FACTOR = 1/Y(I)

FOR ITERATION) 1 SE = 0.695056E-05
FOR ITERATION) 2 SE = 0.222054E-05
FOR ITERATION) 3 SE = 0.101181E-05

.....
.....
.....

FOR ITERATION) 27 SE = 0.684118E-07
FOR ITERATION) 28 SE = 0.678114E-07
FOR ITERATION) 29 SE = 0.672242E-07
FOR ITERATION) 30 SE = 0.666495E-07

THE AREAS UNDER THE VARIOUS PEAKS ARE GIVEN BELOW:
AREAS = 1.21395 2.55963 3.58372 3.76429
INTEGRAL BREADTH = 0.03078 0.10309 0.10572 0.03610
BASELINE AREA = 0.93942

FINAL FITTING PARAMETERS USED

PEAK POS INT AT MAX HALF-WIDTH GAUSS
0 0.13344 0 39.44498 0 0.02477 0 0.65
0 0.18465 0 24.83977 0 0.05563 1 0.00
0 0.27359 0 32.83696 0 0.08143 0 0.54
0 0.29227 0 104.27428 0 0.02861 0 0.61

BASELINE POSITIONS AND INTENSITIES
0.0679 3.00000
0.4437 2.00000

THIS WAS THE MAXIMUM ITERATION BUT IT HAS NOT YET
CONVERGED. DO YOU WANT TO TRY AGAIN;

NO

DO YOU WANT A PRINT-OUT OF THE FITTED DATA;
YES

BEST AVAILABLE COPY

TABLE 3 CONTINUED

THETA	S(1/A)	INTENSITIES			INDIVIDUAL PEAKS	PK 1	PK 2	PK 3	PK 4
		OBSERVED	CALCULATED	OBS-CALC					
6.00	0.0579	7.113	6.821	0.292	3.000	0.536	2.551	0.559	0.165
6.50	0.0735	7.282	7.243	0.039	2.985	0.645	2.851	0.589	0.173
7.00	0.0792	7.220	7.753	-0.533	2.970	0.789	3.190	0.621	0.183
7.50	0.0848	7.521	6.382	-0.860	2.955	0.988	3.590	0.656	0.193
8.00	0.0905	8.724	9.189	-0.465	2.940	1.287	4.065	0.694	0.204
8.20	0.0927	9.396	9.591	-0.195	2.934	1.459	4.280	0.710	0.206
8.40	0.0950	10.201	10.064	0.137	2.928	1.686	4.510	0.727	0.213
8.60	0.0973	10.328	10.637	-0.309	2.922	1.995	4.759	0.744	0.218
8.80	0.0995	11.895	11.357	0.538	2.916	2.430	5.426	0.762	0.223
9.00	0.1018	12.936	12.288	0.648	2.910	3.054	5.315	0.780	0.228
9.20	0.1040	14.040	13.515	0.524	2.904	3.951	5.627	0.799	0.234
9.40	0.1063	15.109	15.143	0.966	2.898	5.232	5.964	0.819	0.240
9.60	0.1085	17.755	17.285	0.468	2.892	6.981	6.328	0.840	0.245
9.80	0.1108	20.754	20.853	0.711	2.886	9.332	6.723	0.861	0.252
10.00	0.1131	23.880	23.522	0.359	2.880	12.350	7.151	0.884	0.258
10.20	0.1153	27.164	27.708	-0.544	2.874	16.048	7.614	0.907	0.264
10.40	0.1176	32.254	32.538	-0.284	2.868	20.351	8.117	0.931	0.271
10.60	0.1198	37.332	37.822	-0.489	2.862	25.064	8.662	0.956	0.278
10.80	0.1221	42.561	43.229	-0.668	2.856	29.853	9.253	0.981	0.286
11.00	0.1243	47.052	48.278	-1.226	2.850	34.234	9.892	1.009	0.293
11.20	0.1266	52.205	52.359	-0.154	2.844	37.593	10.584	1.037	0.301
11.40	0.1288	54.851	54.857	-0.006	2.838	39.313	11.331	1.066	0.310
11.60	0.1311	56.060	55.397	0.663	2.832	39.015	12.135	1.097	0.318
11.80	0.1333	55.081	54.049	1.032	2.826	36.770	12.996	1.129	0.327
12.00	0.1356	52.655	51.289	1.367	2.820	33.053	13.916	1.163	0.337
12.20	0.1378	48.617	47.752	0.865	2.814	28.502	14.891	1.198	0.347
12.40	0.1401	43.875	44.015	-0.140	2.808	23.699	15.916	1.235	0.357
12.60	0.1423	48.234	48.509	-0.274	2.802	19.083	16.988	1.274	0.368
12.80	0.1446	36.259	37.511	-1.252	2.796	14.945	18.077	1.315	0.379
13.00	0.1468	34.604	35.162	-0.558	2.790	11.442	19.181	1.358	0.391
13.20	0.1491	32.770	33.485	-0.715	2.784	8.621	20.273	1.404	0.403
13.40	0.1513	31.861	32.417	-0.556	2.778	6.448	21.322	1.453	0.416
13.60	0.1536	31.550	31.841	-0.291	2.772	4.838	22.297	1.505	0.429
13.80	0.1558	31.228	31.611	-0.383	2.766	3.682	23.159	1.560	0.443
14.00	0.1581	32.043	31.581	0.462	2.760	2.870	23.874	1.619	0.458
14.20	0.1603	31.949	31.621	0.328	2.754	2.304	24.407	1.683	0.474
14.40	0.1626	32.725	31.628	1.097	2.748	1.987	24.731	1.751	0.490
14.60	0.1648	32.571	31.527	1.043	2.742	1.624	24.829	1.825	0.507
14.80	0.1671	32.240	31.276	0.954	2.736	1.414	24.696	1.905	0.525
15.00	0.1693	31.742	30.857	0.885	2.730	1.252	24.338	1.992	0.545
15.20	0.1716	30.943	30.274	0.669	2.724	1.123	23.776	2.087	0.565
15.40	0.1738	30.183	29.547	0.637	2.718	1.015	23.037	2.198	0.586
15.60	0.1760	28.360	28.705	-0.345	2.712	0.924	22.157	2.304	0.609
15.80	0.1783	26.972	27.784	-0.812	2.706	0.845	21.172	2.428	0.632
16.00	0.1805	25.831	26.816	-0.985	2.700	0.776	20.118	2.565	0.658
16.20	0.1828	25.148	25.836	-0.688	2.694	0.715	19.026	2.716	0.684
16.40	0.1850	23.753	24.878	-1.118	2.688	0.661	17.926	2.882	0.713
16.60	0.1873	23.814	23.943	-0.929	2.682	0.613	16.839	3.066	0.743
16.80	0.1895	22.212	23.073	-0.861	2.676	0.569	15.782	3.269	0.775
17.00	0.1917	21.250	22.272	-1.222	2.670	0.531	14.769	3.493	0.810
17.20	0.1940	21.190	21.552	-0.362	2.664	0.496	13.805	3.740	0.846
17.40	0.1962	19.794	20.519	-1.125	2.659	0.464	12.898	4.013	0.885
17.60	0.1984	20.702	20.376	0.326	2.653	0.435	12.046	4.314	0.927
17.80	0.2007	19.735	19.928	-0.192	2.647	0.409	11.256	4.644	0.972
18.00	0.2029	19.991	19.574	0.418	2.641	0.385	10.520	5.008	1.020
18.20	0.2052	20.227	19.314	0.913	2.635	0.363	9.838	5.407	1.072
18.40	0.2074	20.347	19.150	1.197	2.629	0.343	9.207	5.843	1.128
18.60	0.2096	19.842	19.080	0.762	2.623	0.325	8.625	6.320	1.188
18.80	0.2119	20.175	19.103	1.072	2.617	0.308	8.088	6.838	1.253
19.00	0.2141	20.647	19.219	1.427	2.611	0.292	7.592	7.401	1.323
19.20	0.2163	20.843	19.427	1.416	2.605	0.277	7.135	8.011	1.400
19.40	0.2186	20.551	19.726	0.824	2.599	0.264	6.712	8.668	1.483
19.60	0.2208	20.929	20.115	0.814	2.593	0.251	6.323	9.375	1.573
19.80	0.2230	22.153	20.594	1.559	2.587	0.240	5.952	10.132	1.673
20.00	0.2253	22.001	21.161	0.840	2.581	0.229	5.629	10.940	1.781
20.20	0.2275	22.341	21.815	0.526	2.575	0.219	5.321	11.759	1.900
20.40	0.2297	22.875	22.555	0.320	2.569	0.209	5.035	12.709	2.032
20.60	0.2319	23.411	23.380	0.031	2.563	0.200	4.771	13.669	2.177
20.80	0.2342	24.336	24.288	0.048	2.558	0.192	4.525	14.676	2.338
21.00	0.2364	25.393	25.279	0.115	2.552	0.184	4.295	15.729	2.518

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TABLE 3 CONCLUDED

21.20	0.2386	26.443	26.349	0.093	2.546	0.177	4.063	16.824	2.720
21.40	0.2428	26.523	27.500	-0.976	2.540	0.170	3.885	17.956	2.948
21.60	0.2431	27.837	28.729	-0.892	2.534	0.163	3.760	19.122	3.210
21.80	0.2453	29.275	30.041	-0.767	2.528	0.157	3.527	20.315	3.514
22.00	0.2475	30.782	31.441	-0.659	2.522	0.151	3.366	21.528	3.874
22.20	0.2497	31.555	32.940	-1.386	2.516	0.146	3.215	22.752	4.311
22.40	0.2520	33.342	34.561	-1.219	2.510	0.141	3.073	23.978	4.858
22.60	0.2542	35.124	36.336	-1.212	2.504	0.136	2.941	25.196	5.560
22.80	0.2564	37.272	38.320	-1.048	2.498	0.131	2.816	26.392	6.483
23.00	0.2586	40.629	40.588	0.041	2.492	0.127	2.699	27.553	7.717
23.20	0.2608	42.825	43.242	-0.417	2.487	0.123	2.589	28.665	9.379
23.40	0.2631	46.399	46.410	-0.011	2.481	0.119	2.485	29.712	11.614
23.60	0.2653	58.368	50.243	0.125	2.475	0.115	2.387	30.677	14.589
23.80	0.2675	55.692	54.902	0.790	2.469	0.111	2.295	31.544	18.483
24.00	0.2697	59.932	50.540	-0.608	2.463	0.108	2.207	32.296	23.466
24.20	0.2719	68.411	67.274	1.137	2.457	0.104	2.125	32.918	29.670
24.40	0.2741	74.277	75.155	-0.877	2.451	0.101	2.047	33.395	37.160
24.60	0.2763	84.645	84.133	0.512	2.445	0.098	1.973	33.718	45.898
24.80	0.2785	93.717	94.027	-0.309	2.439	0.095	1.903	33.879	55.710
25.00	0.2808	104.308	104.489	-0.181	2.434	0.093	1.836	33.873	66.254
25.20	0.2830	113.834	114.981	-1.148	2.428	0.090	1.773	33.702	76.989
25.40	0.2852	125.052	124.745	0.307	2.422	0.087	1.713	33.369	87.154
25.60	0.2874	132.939	132.816	0.123	2.416	0.085	1.656	32.882	95.777
25.80	0.2896	139.759	138.129	1.630	2.410	0.083	1.602	32.254	101.780
26.00	0.2918	140.434	139.777	0.657	2.404	0.080	1.550	31.498	104.244
26.20	0.2940	139.005	137.351	1.654	2.398	0.078	1.501	30.630	102.744
26.40	0.2962	131.696	131.136	0.559	2.392	0.076	1.454	29.665	97.548
26.60	0.2984	121.864	121.984	-0.120	2.387	0.074	1.409	28.622	89.492
26.80	0.3006	110.804	110.977	-0.173	2.381	0.072	1.367	27.515	79.643
27.00	0.3028	98.621	99.140	-0.519	2.375	0.071	1.326	26.361	69.008
27.20	0.3050	84.959	87.300	-2.341	2.369	0.069	1.287	25.175	58.401
27.40	0.3072	75.466	76.055	-0.589	2.363	0.067	1.249	23.968	48.487
27.60	0.3094	64.304	65.801	-1.497	2.357	0.066	1.213	22.754	39.411
27.80	0.3116	56.389	56.759	-0.370	2.351	0.064	1.179	21.543	31.622
28.00	0.3138	48.743	49.006	-0.263	2.346	0.062	1.146	20.344	25.108
28.20	0.3160	43.729	42.508	1.221	2.340	0.061	1.115	19.165	19.827
28.40	0.3182	39.269	37.154	2.115	2.334	0.060	1.084	18.013	15.663
28.60	0.3204	33.440	32.792	0.649	2.328	0.058	1.055	16.894	12.456
28.80	0.3226	30.013	29.250	0.763	2.322	0.057	1.027	15.812	10.031
29.00	0.3248	27.343	26.364	0.980	2.316	0.056	1.001	14.772	8.219
29.20	0.3270	25.286	23.986	1.300	2.311	0.054	0.975	13.776	6.871
29.40	0.3292	22.806	21.998	0.808	2.305	0.053	0.950	12.827	5.863
29.60	0.3314	21.230	20.303	0.927	2.299	0.052	0.926	11.927	5.099
29.80	0.3335	19.810	18.831	0.979	2.293	0.051	0.903	11.075	4.509
30.00	0.3357	18.153	17.532	0.621	2.287	0.050	0.881	10.274	4.040
30.20	0.3379	16.222	16.369	-0.147	2.281	0.049	0.860	9.522	3.657
30.40	0.3401	15.076	15.319	-0.243	2.276	0.048	0.839	8.820	3.337
30.60	0.3423	14.172	14.364	-0.192	2.270	0.047	0.819	8.165	3.063
30.80	0.3445	13.064	13.492	-0.428	2.264	0.046	0.800	7.558	2.825
31.00	0.3467	12.328	12.695	-0.367	2.258	0.045	0.782	6.995	2.615
31.20	0.3488	11.213	11.964	-0.752	2.252	0.044	0.764	6.476	2.428
31.40	0.3510	10.812	11.296	-0.484	2.247	0.043	0.747	5.998	2.261
31.60	0.3532	10.055	10.683	-0.528	2.241	0.042	0.730	5.553	2.111
31.80	0.3554	9.733	10.123	-0.390	2.235	0.042	0.714	5.158	1.975
32.00	0.3575	8.994	9.610	-0.616	2.229	0.041	0.698	4.790	1.852
32.20	0.3597	8.597	9.141	-0.544	2.223	0.040	0.683	4.455	1.739
32.40	0.3619	7.842	8.712	-0.871	2.218	0.039	0.669	4.150	1.637
32.60	0.3641	7.703	8.321	-0.617	2.212	0.039	0.655	3.872	1.543
32.80	0.3662	7.337	7.962	-0.625	2.206	0.038	0.641	3.620	1.457
33.00	0.3684	7.281	7.634	-0.354	2.200	0.037	0.628	3.391	1.379
33.50	0.3738	6.236	6.931	-0.695	2.186	0.036	0.596	2.906	1.207
34.00	0.3793	6.161	6.363	-0.202	2.171	0.034	0.567	2.523	1.065
34.50	0.3847	5.745	5.901	-0.157	2.157	0.033	0.540	2.224	0.947
35.00	0.3901	5.324	5.520	-0.196	2.143	0.031	0.515	1.983	0.848
35.50	0.3955	5.095	5.201	-0.106	2.128	0.030	0.492	1.787	0.763
36.00	0.4008	4.852	4.929	-0.277	2.114	0.029	0.470	1.625	0.691
36.50	0.4062	4.867	4.695	0.173	2.100	0.028	0.450	1.489	0.628
37.00	0.4116	4.395	4.489	-0.094	2.085	0.027	0.431	1.373	0.574
37.50	0.4170	4.597	4.307	0.290	2.071	0.026	0.413	1.271	0.526
38.00	0.4223	4.764	4.144	0.620	2.057	0.025	0.396	1.182	0.484
38.50	0.4277	4.576	3.997	0.579	2.043	0.024	0.381	1.103	0.447
39.00	0.4330	4.591	3.863	0.728	2.028	0.023	0.366	1.031	0.414
39.50	0.4363	4.567	3.740	0.827	2.014	0.022	0.352	0.967	0.385
40.00	0.4437	4.596	3.628	0.968	2.000	0.022	0.339	0.909	0.359

DO YOU WANT A PLOT? TYPE Y OR N

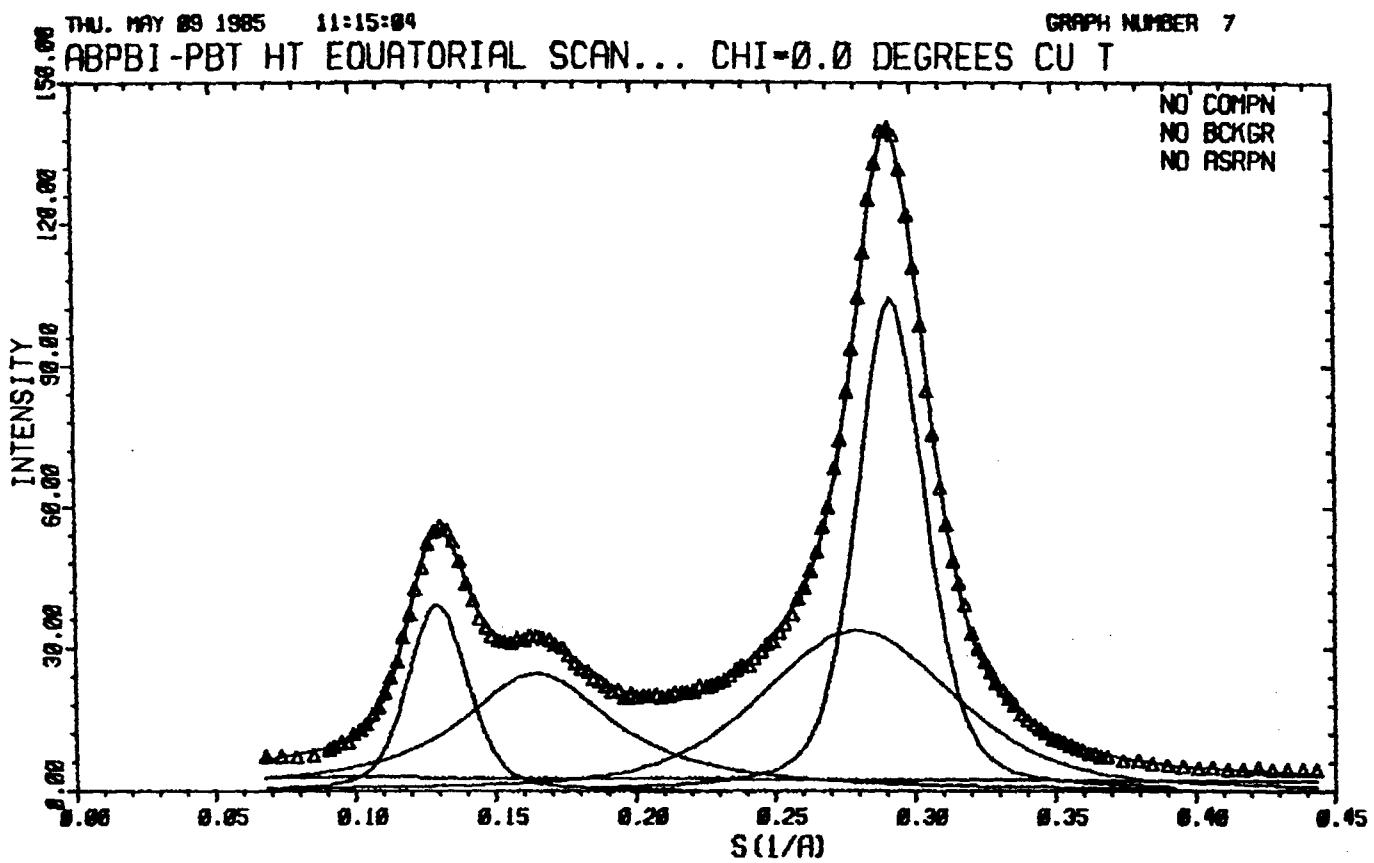
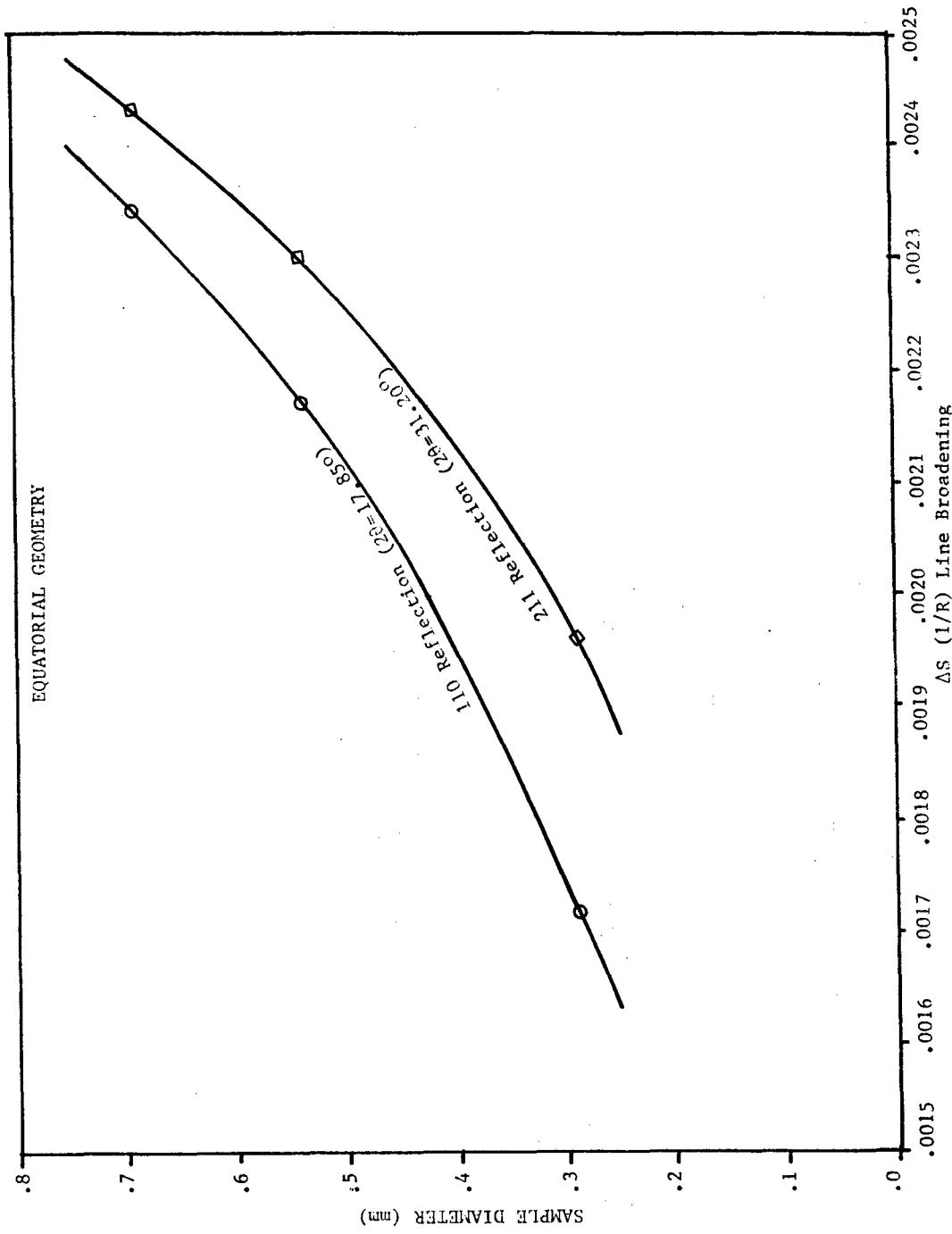
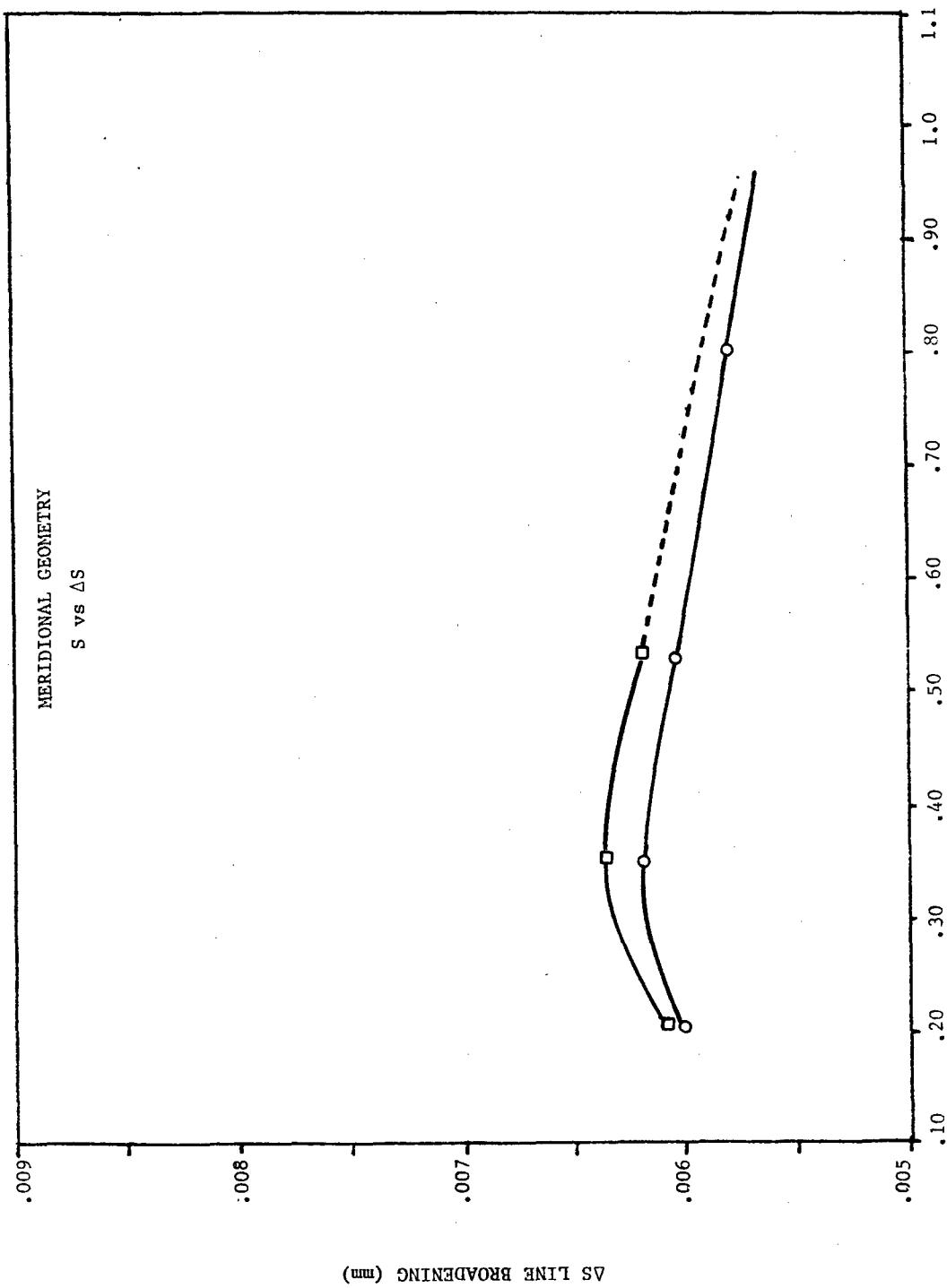


Figure 4. Graph of ABPB1-PBT Blend Equatorial Scan. Cu K α with monochromator. Solid curves show calculated peak profiles for the four individual peaks and the sum. Parameters as in table 3.



a. Equatorial geometry, sample diameter vs Δs for reflections at 17.85° and 31.20° 2θ .

Figure 5. Plots of Full Width at Half Maximum (in units of $2\sin\theta/\lambda$) Standards.



b. Meridional geometry, S vs Δs , data for 0.29 mm and 0.54 mm capillaries.

Figure 5 Concluded

SECTION V

ORIENTATION FACTOR MEASUREMENTS: RESOLVED PEAKS

Orientation of the crystallites in polymer materials is a significant feature in the characterization of polymers. The degree of orientation is commonly expressed in terms of Herman's orientation factor (Stein, 1958), the calculation of which requires the intensity of a reflection (usually either equatorial or meridional) expressed as a function of X . In the equatorial case the orientation factor is given by

$$f = \frac{\sum I (3 \sin^2 X - 1) \cos X \Delta X}{2 \sum I \cos X \Delta X},$$

where I is the intensity, X is the diffractometer angle and ΔX is the step size. If the diffraction peak one uses is resolved, its intensity can be determined by a simple scan with background measured at each end of the scan.

The following experiments were done and the results obtained using (unless specified otherwise) the substances and measurement parameters listed below:

Sample	Heat-treated PBT fibers
x-ray source	Cu standard focus Cu tube (take-off angle of 3.0°) with HOG monochromator set at 2θ of 26.76° and PSI of 13.55° .
Collimation	1 mm-diameter collimators with diffracted beam (SVA) aperture of 3 mm by 3 mm.
Analyzer	Gain at 10, rejection limits upper 7.0, lower 4.0. Photomultiplier voltage potentiometer set at 4.42 (about 950 volts).

MEASUREMENT OF INTEGRATED INTENSITY AND ALIGNMENT OF THE SAMPLE

To measure the integrated intensities required for the orientation factor calculation, one first examines a 2θ scan, selecting the 2θ peak position and the background positions above and below the peak ($\pm \Delta 2\theta$). The integrated intensity is then the peak count rate minus the average background count rate. This measurement is conveniently made by using the /FB command mode of the FACS-I Control Program.

Preliminary scans should be used to establish that the sample is aligned on the diffractometer so that X scans are symmetrical about $X = 0^\circ$ for equatorial scans and about $X = 90^\circ$ for meridional scans. This is most conveniently done by scanning from negative to positive X values. Steps of 1.0° in X will give a profile satisfactory for this purpose. The goniometer head should be adjusted to improve alignment and the scans repeated until the profile is symmetrical.

CHI RANGE

Next determine the point in χ where the intensity reaches background. The following example shows a typical scan with apparent intensity beyond the tails of the peak. The scan must be terminated at the point where the intensity drops to zero and any non-zero intensity measurements beyond must be eliminated from the orientation factor calculation. Since the program used for the calculation assigns a zero intensity to all points not measured, the χ scan should be terminated at the point where intensity from the peak of interest drops to zero. See tables 4 and 5.

TABLE 4. EXAMPLE PRINTOUT CHECK FOR TAIL OF A PBO DISTRIBUTION. THE SCAN RUNS FROM CHI 20 TO 30 DEGREES; COUNT RATES IN CPS. COUNTS AT $\chi = 0^\circ$ APPROXIMATELY 170 CPS.

/PT

6

/FB

5 8

/PO

MTHD=1

TH=16 TH=0

OM=0 DLSN=0 NPTS=1 CH=20 DLCH=1 NPTS=11

TH= 16.00 OM= 0.000 CG= 20.00 PH= 0.00

OM= 0.00

3.00 2.45 0.93 1.07 1.27 1.65 1.13 1.10 0.97 1.40

0.02

TABLE 5. EXAMPLE ORIENTATION FACTOR RUN FOR PBO (AS-SPUN) #042083N1

/PT

8

/FB

7 8

/PO

MTHD=1

TH=16 TH=0

OM=0 DLSN=0 NPTS=1 CH=0 DLCH=.5 NPTS=55

TH= 16.00 OM= 0.00 CH= 0.00 PH= 0.00

OM=

173.83	172.62	167.61	160.15	149.30	139.16	123.85	111.85	98.80	87.73
76.67	66.41	58.22	49.45	42.04	37.89	32.68	28.59	24.67	20.89
18.76	15.85	13.45	12.40	11.35	9.99	8.78	7.22	7.69	5.72
5.57	5.19	4.29	3.41	3.79	3.58	3.36	2.91	2.65	1.57
2.80	3.04	1.86	1.78	1.33	0.79	1.57	2.46	1.02	1.74
1.04	-1.72	3.67	0.83	0.76					
16.5464		-0.4848							

CHI STEP SIZE

When the sample alignment is satisfactory and the χ range has been determined, the step size for χ can be investigated. Note that highly oriented samples require careful alignment, a smaller χ range and a smaller step size for χ . The step size has been investigated by scanning a set range in χ with different step sizes. Since the PBT fibers used are fairly highly oriented (equatorial reflections have a full width at half maximum for χ of about 8°), step sizes of 1.5° , 1° and 0.5° were used. The number of points measured was increased so as to include the same range of χ in each experiment. In all cases the integrated intensity was taken to be $I_p - I_b$, where I_p and I_b represent the count rate at the peak position in 2θ and the background (offset by $\pm 2\theta$). As explained above, the intensity of the reflection being examined was taken as zero outside the χ range actually scanned.

The results show a possible tendency for the orientation factor to decrease with decreasing step size. The orientation factor, obtained for the $\chi = 0^\circ$ to 10° range are in table 6.

TABLE 6. ORIENTATION FACTORS FOR TWO PBT REFLECTIONS. CHI STEPS OF 1.5° , 1.0° , 0.5° .

2θ	$\Delta\chi$	Orientation Factor
15.15	1.5°	-.4964
15.15	1.0°	-.4964
15.15	0.5°	-.4961
25.4	1.5°	-.4957
25.4	1.0°	-.4957
25.4	0.5°	-.4957

INSTRUMENTAL FACTORS

For highly oriented materials, the width of the peak for a χ scan, and hence the orientation factor, will be affected by instrumental factors such as source, sample and detector sizes. The detector aperture size, which is easily changed, gives an indication of the magnitude of the instrumental broadening. The scans at $25.4^\circ 2\theta$ were repeated with a detector height (vertical dimension) set at 3 mm, 2 mm and 1 mm. The results are in table 7.

TABLE 7. ORIENTATION FACTORS FOR VARIOUS SVA HEIGHTS.

2θ	$\Delta\chi$	SVA height	Orientation Factor
25.4	1.0°	3 mm	-0.4957
25.4	1.0°	2 mm	-0.4957
25.4	1.0°	1 mm	-0.4957

Thus we conclude that for the degree of orientation seen here, the instrumental factors do not affect the orientation factor.

PRECISION OF THE ORIENTATION FACTOR

The precision of the orientation factor, as determined by the techniques described above, was also investigated by repeated scans. The results are in table 8.

TABLE 8. PRECISION OF ORIENTATION FACTOR

2θ	$\Delta\chi$	Orientation Factor	Average Orientation Factor	Sigma Average
15.15°	1.0°	-0.4964		
		-0.4965		
		-0.4964	-0.49642	0.00002
		-0.4964		
		-0.4964		
15.15°	0.5°	-0.4962		
		-0.4962		
		-0.4961	-0.49610	0.00005
		-0.4960		
		-0.4960		
25.4°	1.0°	-0.4957		
		-0.4958		
		-0.4956	-0.49568	0.00004
		-0.4957		
		-0.4956		

For the results in table 8, the peak intensities ($I_p - I_b$) were about 100 cps, and measurement times were about 100 s for the peak and 40 s for each of the two background measurements. In all of the above, σ for a single observation is about 0.0001. We conclude, based on these results, that for samples with orientation factors from about -0.496 to zero, a step size of 0.5° in χ and counting times which give individual observations with $\sigma(I)$ of about 1% will provide orientation factor measurements with a statistical accuracy of 0.0001.

SECTION VI

DETERMINATION OF ORIENTATION FOR POLYMER FIBERS:

OVERLAPPING REFLECTIONS

Composite materials such as PBT-ABPBI blends may contain crystallites of both components. Heat-treated PBT-ABPBI blends apparently contain both PBT crystallites and ABPBI crystallites, and the PBT and ABPBI crystallites may differ as to degree of orientation. Both PBT and ABPBI have prominent peaks correspondent to 3.5\AA spacing. In addition, PBT has a peak at 5.9\AA and ABPBI has one at 7.0\AA . Since the peaks needed to characterize the orientation are unresolved neither the peak height nor the intensity of the reflection can be obtained directly from the diffractometer scan. The procedure developed to handle this case is described below. It involves 2θ scans taken at intervals in χ . A least-squares procedure is used to fit the overlapping peaks in each 2θ scan and thus resolve the peaks so that the peak area for each peak of interest can be obtained as a function of χ .

SELECTION OF INSTRUMENT AND SCAN PARAMETERS

The first step in treating the case of unresolved peaks is to select data collection conditions that give the best resolution obtainable for the peaks of interest. This involves a selection of the x-ray tube take-off-angle, the diffracted beam aperture, the radiation and the monochromator or filter to use. Smaller take-off-angles and apertures give better resolution but also reduce the count rate, and, therefore, the statistical accuracy of the measurements, unless counting times are increased to compensate.

The next step is to determine the scan parameters. Two-theta scans must be made at intervals in χ and, for highly crystalline material, the 2θ step size must be small to ensure that the peak profile is accurately recorded. In regions between the peaks of interest, the step size can be larger. Note that all peaks in the scan range must be recorded in sufficient detail to give an accurate peak profile or they must be omitted entirely. This is because the set of peaks used by the least-squares program, usually only the peaks of interest, must match the experimental curve in order to obtain a satisfactory fit. The /TH command can be used to define the 2θ range and select the step size for the different regions of the scan. For highly oriented material the increments must not be too large. There should be a minimum of 4 or 5 scans that show a contribution from the peaks of interest. If there is clearly no contribution beyond a certain point in χ , scanning can be omitted beyond that point.

When the data scans have been completed, you are ready to measure the background. Since the background is a function of 2θ , the background scan must include measurements at all 2θ points included in the data scans. Contributions from diffraction peaks or layer lines must, of course, be avoided. The best scan to use for the background correction can usually be selected by examining a diffraction photograph of the material under study. Mount the negative on a film-measuring device so that θ and χ can be conveniently determined. Find the value of χ which gives the 2θ scan with the least interference from layer lines and make the background /TH scan for that χ . Note that the χ interval between each /TH scan should be the same

and each $/TH$ scan must have measurements for the same set of 2θ values.

DATA PROCESSING

When the data have been measured and written on the magnetic tape, translated and entered into the off-site computer, you are ready to process it for input to the curve fitting program. This program subtracts the background scan from the other scans point-by-point, makes the LP correction, and writes a file in a format corresponding to the input format required by the Anderson peak fitting program (Anderson 1985). The preprocessing program has been affectionately named GALEN by one of us. Although the name is not very descriptive, it is better than his usual porcine choices.

In the present example, fibers of the heat-treated PBT-ABPBI blend were scanned at values of 0° , 4.5° , 9.0° , 13.5° , 18.0° , 22.5° , 27.0° , and 31.5° . Inspection of the diffraction pattern of a similar fiber showed that one feature on the 3.15° scan was due to the first layer line. The intensity values for that region were replaced by inserting a straight-line interpolation in place of the original intensities. The data file was then run through the preprocessing program.

Two theta scans for the lower χ values show three peaks. Two at about 10 degrees 2θ (Cu K α data) are barely resolved and a third at about 25 degrees 2θ appears to be resolved but is actually the sum of two peaks, one from each component of the blend. Samples of heat-treated PBT and heat-treated ABPBI photographed separately show that the 25-degree peak is present in both. Preliminary attempts to fit the heat-treated blend with three peaks resulted in a poor fit in the region between the doublet and the main peak. It was found that a good fit could be obtained by assuming that the 25-degree peak was the sum of two peaks with different width parameters.

Apparently one component of the blend has fairly sharp peaks at 10 degrees and 25 degrees and the other component has broader peaks at 11 degrees and 25 degrees. Peak parameters are best defined on the scans at $\chi = 0^\circ$; therefore the peak positions obtained on those scans should be used for fitting the other scans. The peak areas obtained at each χ value can be used in the usual formula to calculate the orientation factor for each component. The same orientation factor should be obtained for each of the two peaks arising from the same component. If interference from another layer line is present, the peak parameters will be inaccurate and that scan cannot be used. It must then be replaced by an estimated peak area, otherwise the orientation factor will be incorrect. A better solution to the layer line problem would be to subtract the intensity due to the interfering layer line before running the preprocessing program. When this is done the peak parameters obtained by the fitting program should be accurate. If any peak height, area or width is negative, you have a problem.

SECTION VII

DIFFRACTOMETER SCANS ALONG LAYER LINES FOR POLYMER MATERIALS

Polymer fibers give diffraction patterns which show cylindrical symmetry with the diffraction maxima distributed in a series of layer lines. In highly ordered specimens, the layer lines are made up of individual reflections or groups of reflections.

It is convenient to visualize the fiber pattern as it would appear if recorded on a precession film. If the fiber is vertical, the layer lines on the film will be horizontal and a raster scan of the film will have one scan direction along the layer lines. The intensity variation along the layer lines will be gradual for materials of moderate order but will show sharp changes for highly ordered materials.

The c^* axis is, by convention, assigned to the layer line repeat which is parallel to the fiber axis, and the layer lines are then designated by the l index. Because of the cylindrical symmetry of the pattern, either h or k can be used to designate points along each of the layer lines.

The automated diffractometer can be used to measure the intensity along the layer lines. This is conveniently done by using the single crystal routines of the diffractometer control software. The method, as coded, requires an orientation matrix and cell constants for a unit cell with c^* parallel to the ϕ axis of the diffractometer. Thus any fiber bundle that can be mounted on the diffractometer with the fiber axis parallel to the ϕ axis is suitable for a layer line scan. The only angles varied during the scan are 2θ and χ , so the sample mount is not required to allow ϕ rotation except for optical centering and alignment.

The first step in preparing for a layer line scan is to center the fiber and ensure that the fiber is exactly parallel to ϕ . If the specimen has sharp reflections on the meridian, one of these may be used. The goniometer head arcs must be adjusted so that the reflection is centered in the detector aperture at $\chi=90^\circ$ and $\omega=0^\circ$.

A convenient procedure for setting the fiber axis parallel to the axis of the diffractometer follows:

1. Use the diffractometer telescope to center the sample in the x-ray beam and align it as nearly as possible with the ϕ axis.
2. With $\chi=90^\circ$, rotate the sample so that one goniometer arc is vertical (in the plane of the χ circle).
3. Determine the profile of the strongest sharpest meridional reflection present by making counts at appropriate intervals of χ . For broad peaks, you may use χ steps of 5° ; for sharp peaks, smaller steps will be required. The scan should extend at least $2/3$ of the way down each side of the peak.
4. Adjust the vertical goniometer arc so the peak will be centered at $\chi=90^\circ$.

5. Rotate the goniometer head and repeat steps 1 through 4 with the other arc in the vertical position. Adjust it as required.

Note: The /PO command (see DIFF manual for details) can be used to obtain the X scans or you can drive X and use /PT and /ST to measure each point individually.

The procedure is similar if you have no suitable meridional reflection and must use an equatorial reflection. For an equatorial reflection X must be set to zero.

The fiber repeat spacing must be accurately known. To find it, locate a strong meridional reflection and scan it with the /TH command after setting up a peak fit request with the /FT command. Use the layer line number and the d spacing calculated by the peak fit procedure to find the c axis repeat. You are now ready to set up the cell constants and the orientation matrix.

Enter the cell constants with the /RP command. If the unit cell for the material being studied is not known, use $a = b = c$ and $\alpha = \beta = \gamma = 90^\circ$ (you have determined c). The orientation is specified by two orientation reflections. The first must be an $h00$ reflection with ω , X , and ϕ set to zero, the second an $0k0$ reflection with ω and X at zero and $\phi = 90^\circ$. Calculate the orientation matrix with /CM and display it with /CA after setting the wavelength with /WV.

You are now ready to choose the scan parameters for the layer line scan. If you have a precession film, you can use it as a guide. The layer lines are easily counted to obtain the scan limits which are denoted as the ℓ index. The length of scan along each layer line is specified by a start and stop value for k . If you have used $a = b = c$ for the cell constants, each unit of k will span a length equal to the distance between each layer line on a precession film.

Enter the preset time with /PT to be used for each point measured and then enter the scan limits with /LL. The interval for k will be governed by the degree of order in your sample. Values of 0.2 and 0.5 are often used. Note that since b is arbitrary, the k scan parameters are not related to the real k index. The interval for ℓ is usually 0.5 so that a background scan is included between each layer line.

Table 9 lists data for a first layer line scan on ABPBT fibers, which are plotted in figure 6.

TABLE 9. EXAMPLE OF FACS-I INPUT-OUTPUT FOR A LAYER LINE SCAN FOR A HEAT-TREATED ARPBT SAMPLE.

/WV

1.5418

/PT

7

/RP

12.19 12.12 12.19 0 0 0 5 0 0 0 0 0 0 5 0 0 0 90

/CM

/CA

12.19001 12.18999 12.19000 0.00000 0.00000 0.00000

-0.0820344 0.0000000 0.0000000

0.0000000 -0.0820345 0.0000000

0.0000000 0.0000000 -0.0820345

1.541799 1811.39

/FT

0

/LL

0 1 1 -5 .1 5

0.00 -5.00 0.00

0.69 0.59 0.56 0.63 0.65 0.67 0.75 1.09 1.47 2.36 2.93

3.65 6.90 17.78 28.66 28.25 21.20 12.47 5.89 3.16 2.38 1.78

1.66 1.61 1.48 1.55 1.55 2.36 4.11 15.47 25.45 6.15 2.26

1.39 1.20 1.19 1.14 1.15 1.24 1.29 1.33 1.35 1.55 2.04

2.49 3.27 6.50 4.99 0.29 0.22 0.22 0.26 0.33 5.01 6.65

3.06 2.27 1.74 1.51 1.30 1.21 1.00 1.13 1.25 1.18 1.35

1.32 1.50 2.13 6.28 26.13 15.89 3.98 2.17 1.74 1.65 1.59

1.45 1.73 1.81 2.29 3.27 6.10 11.95 20.73 28.21 28.90 17.15

7.23 3.62 3.09 2.58 1.73 1.09 0.73 0.77 0.59 0.65 0.56

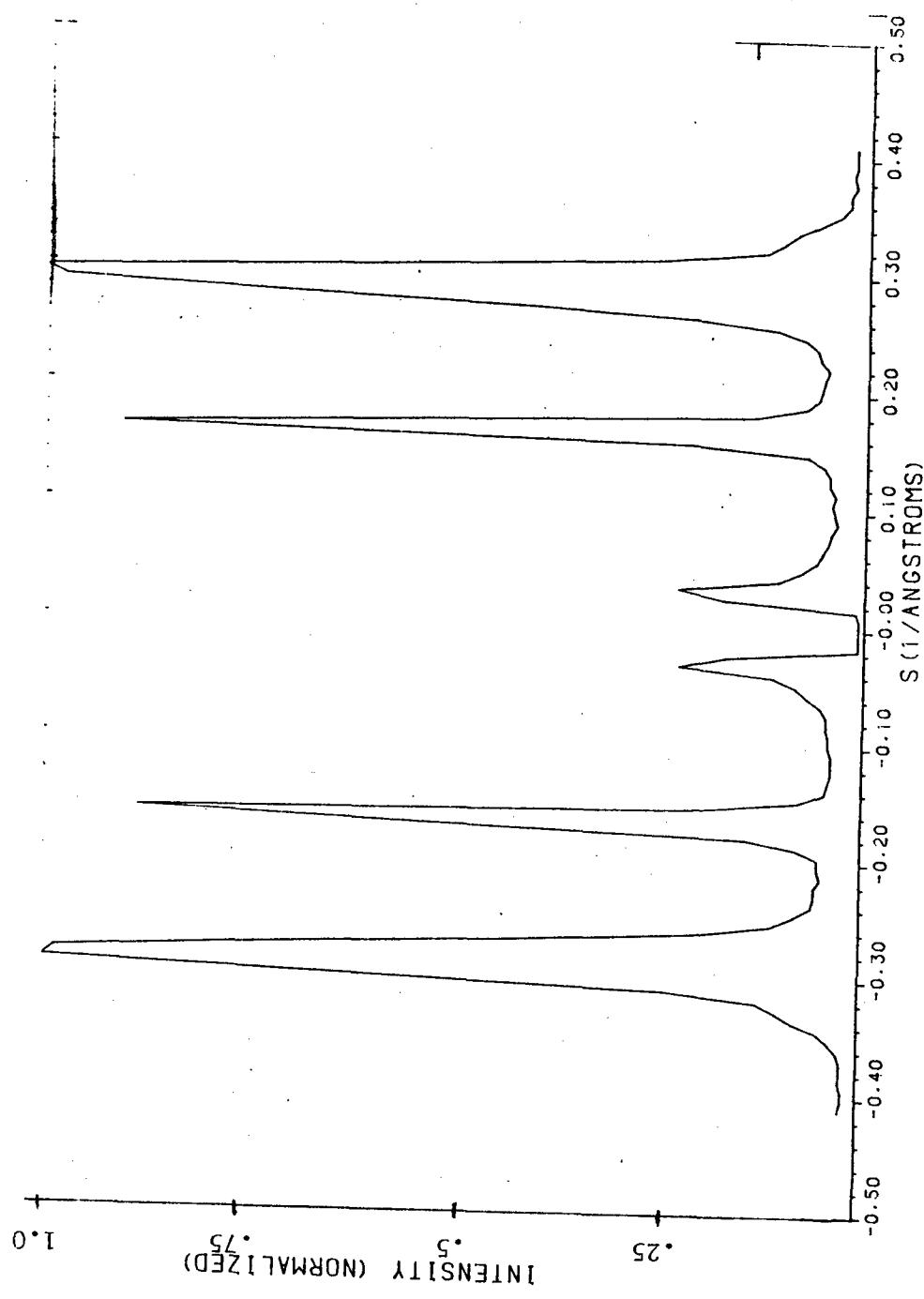


Figure 6. Plot of First Layer Line for ABPBT Fibers.

SECTION VIII

POLE FIGURE DATA COLLECTION AND ANALYSIS

The Picker FACS-I automated diffractometer is ideally suited to collect two-dimensional diffraction data on polycrystalline specimens. These data can be used to determine and display the orientation of the crystallites in the sample in the form of a pole figure diagram. The two diffraction geometries in common use, reflection and transmission, are shown in figure 7 (Desper, 1969).

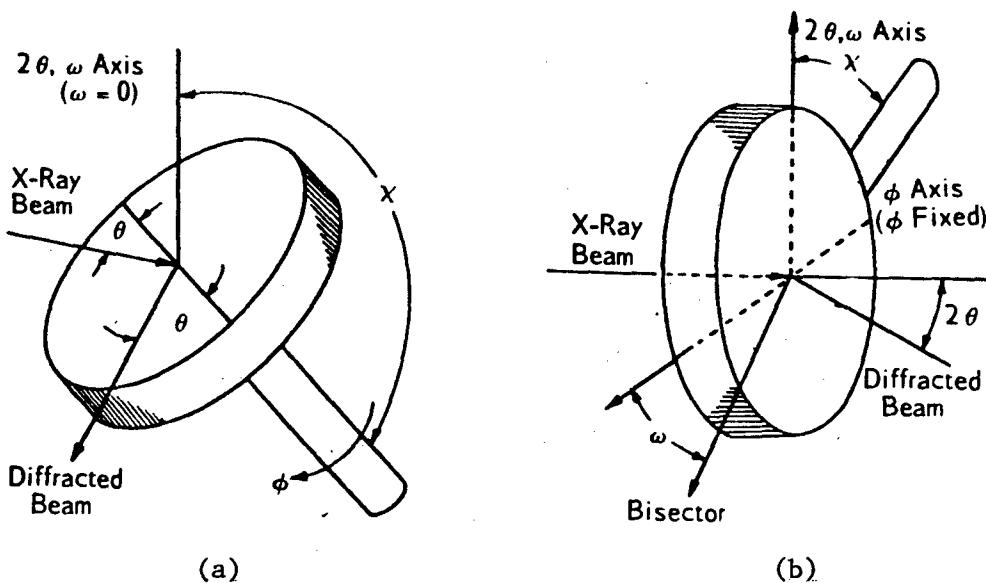


Figure 7. Pole Figure Geometry, (a) Reflection and (b) Transmission Methods

One cannot measure a complete pole figure in either the transmission or the reflection mode selected by MTH0=1 or MTHD=0 in the FACS-I PO command. In the transmission mode all values of χ are available on the diffractometer but ω is restricted to angles between 0 and 45° . This range allows about $2/3$ of the pole figure to be measured. In the reflection mode all values of ϕ are available but there may be mechanical restrictions on values of χ near 0° . At low 2θ angles the x-ray beam becomes almost parallel to the sample even at moderate values of χ . The FACS-I control program includes commands for pole figure data collection which are an extension of those described by Desper (1969).

When starting a pole figure run, be sure to write all instrument parameters on the magnetic tape after initiating it with /TS. Example input is given in table 10 below.

TABLE 10. EXAMPLE OF TAPE HEADING.

/TS

/TY

Data for PBT-27554-33-4(2)

This sample is a bundle of fibers approximately 0.6 mm in diameter. It is mounted vertical in the diffractometer. The scan in 2 Theta will therefore be a scan of the equatorial reflections.

TOA is 3.2DEG (Nominal), Actual TOA is 3.0 DEG.

SVA is wide open.

HOG monochromator XTAL is in place and Beam uniformity is satisfactory (just checked).

Mono angle is 12.16 and PSI is 6.05.

Incident and diffracted beam collimators are 1.0 mm.

No filters are used.

KV 50 MA 12.

Analyzer upper 7.0, Lower 4.0, 100% HT centered at 5.20.

Gain at 10, fine focus molybdenum tube.

Smaller SVA and smaller TOA do not seem to improve the resolution.

The transmission mode is most useful for polymer samples. Job parameters for a transmission mode example are shown in table 11.

TABLE 11. TRANSMISSION MODE POLE FIGURE INPUT AND OUTPUT.

```

/PT
6
/FP
5 3.2
/PO
MTHD=1
TH=7.35 TH=0
OM=0  DLSN=.1  NPTS=8  CH=0  CLCH=2.5  NPTS=37
TH= 7.35  OM= 0.00  CH= 0.00  PH= 0.00

OM= 0.00
10.27 11.95 12.62 11.67 10.32 7.32 5.40 4.40 2.00 1.05
  0.65  0.60  0.27  0.55  0.05  0.35 -0.18  0.15 -0.25  0.10
 -0.10 -0.05 -0.07  0.17  0.25  0.13  0.38  0.40  0.15  0.20
  0.00  0.05 -0.10 -0.05 -0.40 -0.28 -0.28

OM= 5.74
10.47 11.42 11.57 11.12 9.00 7.05 4.92 2.55 1.55 1.05
  0.63  0.55  0.55  0.43  0.32  0.20 -0.05  0.00 -0.17 -0.25
  0.03 -0.10  0.25  0.22  0.30  0.35  0.38  0.42  0.17  0.15
 -0.05  0.15  0.07 -0.15 -0.30 -0.25 -0.40

OM= 11.54
10.70 11.85 12.02 10.97 8.77 6.57 4.40 2.83 1.47 0.77
  0.45  0.32  0.13  0.28  0.02  0.03  0.03  0.03  0.00 -0.13
  0.07 -0.07  0.07  0.20  0.17  0.43  0.43  0.25  0.38  0.35

OM= 17.46
11.12 12.25 11.75 10.70 9.07 6.70 4.35 2.70 1.58 0.82
  0.53  0.57  0.15  0.28  0.20  0.30 -0.07  0.10  0.13  0.07
 -0.20 -0.20 -0.03  0.17 -0.13  0.18  0.28  0.50  0.60  0.45
  0.35  0.30  0.30  0.30  0.05  0.13  0.18

OM= 23.58
11.00 12.12 12.20 11.32 9.15 6.65 4.55 2.77 1.75 0.85
  0.77  0.48  0.50  0.25  0.53  0.20  0.40 -0.05  0.07 -0.15
 -0.05 -0.43 -0.10 -0.03  0.05  0.18  0.15  0.45  0.50  0.53
  0.63  0.50  0.70  0.53  0.45  0.40  0.38

OM= 30.00
12.20 12.72 12.67 11.42 9.47 7.65 5.15 3.40 1.95 1.10
  0.50  1.30  0.60  0.73  0.63  0.73  0.50  0.25  0.32  0.05
 -0.20 -0.13 -0.10 -0.27 -0.10 -0.10 -0.05  0.17  0.10  0.22
  0.35  0.63  0.70  0.57  0.75  0.75  0.75

OM= 36.87
15.65 17.45 17.45 17.10 14.98 12.35 10.35 7.65 5.85 3.77
  2.42  1.67  1.28  0.77  0.85  0.47  0.68  0.63  0.35  0.20
  0.07  0.15 -0.25 -0.23 -0.13 -0.27 -0.10 -0.13 -0.15  0.10
  0.15  0.28  0.30  0.57  0.60  0.57  0.83

OM= 44.43
17.92 19.80 19.72 19.00 17.58 15.45 12.97 10.23 7.80 5.80
  3.95  2.72  1.93  1.47  1.18  0.63  0.77  0.57  0.50  0.33
  0.18  0.80  0.75  1.25  1.80  3.03  3.52  4.55  5.40  6.25
  6.75  7.85  8.18  8.53  8.72  9.55  9.53

```

The intervals in this example for ANGLE 1 and ANGLE 2 (ω and X) are satisfactory for most polymer work. Smaller increments would be advisable if the sample were highly crystalline. Counting time should be chosen so that values near the peak will have at least 2000 counts per measurement. If time permits, more counts will give an improved pole figure in most cases. If a background correction is made, the total time spent counting the background should be about equal to that spent measuring the peak.

The scan range (for the integral mode) or the width of the peak (for the /FB mode) should be carefully chosen. A 2θ scan using the /TH command will give the peak profile and facilitate the choice of optimal end points for the scan. Include all of the peak if possible, but stay clear of adjacent peaks. An example of /TH use is given in table 12.

TABLE 12. TWO-THETA SCAN FOR A BUNDLE OF PBT FIBERS. (PART OF OUTPUT IS OMITTED).

/TY

START 2 THETA EQUATORIAL SCAN AT 5 PM AUG. 4, 1980

/TH

TH=29.	DLTH=.1	TH=29.	DLTH=0							
4.66	4.71	4.65	4.52	4.61	4.65	4.72	4.91	4.88	4.93	
5.02	4.96	4.98	4.83	5.05	5.03	5.11	5.28	5.28	5.28	
5.36	5.39	5.60	5.82	5.72	5.86	5.95	6.19	6.26	6.37	
6.36	6.75	6.85	7.01	7.15	7.34	7.61	7.92	8.13	8.34	
8.70	8.75	8.90	9.36	9.45	9.80	9.82	10.06	10.28	10.53	
10.66	10.78	11.04	11.24	11.47	11.25	11.62	11.60	11.59	11.50	
11.65	11.35	11.45	11.28	11.34	11.09	10.86	10.43	10.55	10.23	
10.01	9.85	9.55	9.53	9.36	9.04	8.89	8.77	8.67	8.50	
8.18	8.24	8.24	8.05	7.88	7.80	7.79	7.60	7.73	7.62	
7.48	7.31	7.42	7.29	7.24	7.14	7.19	6.98	6.98	6.98	
7.08	6.84	6.82	6.76	6.91	6.79	6.76	6.69	6.72	6.43	
6.50	6.58	6.50	6.41	6.57	6.39	6.48	6.57	6.52	6.70	
6.52	6.65	6.72	6.75	6.87	7.01	6.89	7.02	7.27	7.33	
7.44	7.50	7.78	8.04	8.34	8.38	8.85	9.28	9.64	10.23	
10.73	11.48	12.17	13.02	14.04	15.25	16.75	18.39	19.91	21.96	
24.20	26.55	29.17	32.11	35.44	38.67	42.67	46.86	50.94	56.63	
62.26	67.38	73.57	80.16	85.86	91.93	96.62	102.00	105.38	107.99	
110.02	110.55	110.68	109.62	107.95	105.15	100.88	96.01	92.36	86.70	
81.47	75.69	70.47	65.12	61.01	56.41	53.04	50.27	47.34	45.49	
43.57	42.39	40.98	39.88	38.93	38.26	37.90	37.22	36.64	36.84	
36.67	36.53	37.01	37.28	37.72	38.36	39.07	39.68	41.18	42.53	
43.71	45.26	46.69	48.65	49.76	50.56	51.61	51.82	51.85	51.80	

A satisfactory run could have been obtained for this material with a larger step size. A shorter counting time (40s rather than 100s) would also have been satisfactory. The scan was run from -2θ to $+2\theta$ which is desirable but not necessary.

Parameters for UNIAXIAL scans for this PBT fiber bundle are shown in tables 13 and 14.

TABLE 13. UNIAXIAL SCANS FOR PBT. NO BACKGROUND NECESSARY.

```
/PT
6
/PM
/PO
MTHD=1
TH=7.15    TH=0
OM=0      DLSN=0      NPTS=1      CH=0      DLCH=2.5      NPTS=37
TH=    7.15  OM=    0.00  CH=    0.00  PH=    0.00
OM=    0.00
57.25  48.75  37.80  28.85  21.65  16.77  13.70  12.15  11.27  10.60
10.20  9.70   9.20   8.52   8.00   7.45   7.25   6.72   6.67   6.88
  6.85  6.88   7.05   6.90   6.92   6.63   6.55   6.38   6.02   5.77
  5.42  5.60   5.25   5.07   5.07   5.10   5.10
```

TABLE 14. UNIAXIAL SCAN FOR PBT WITH BACKGROUNDS MEASURED FOR 20 S EACH, 1.3° ABOVE AND BELOW THE PEAK POSITION.

1.3° above and below the peak position.

```
/TY
NOW DO IT AGAIN WITH BACKGROUND SUBTRACTED
/FB
5 2.6
/PO
MTHD=1
TH=7.15    TH=0
OM=0      DLSN=0      NPTS=1      CH=0      DLCH=2.5      NPTS=37
TH=    7.15  OM=    0.00  CH=    0.00  PH=    0.00
OM=    0.00
22.70  18.28  13.17  8.15   4.73   2.83   1.50   1.25   0.55   1.25
  0.23  0.85   0.33   0.70   -0.38  -0.02  -0.33  -0.25  0.05  -0.17
  0.20  0.67   0.47   1.12   1.38   1.27   1.10   1.05   0.77   0.75
  0.47  0.63   0.55   0.40   0.40   0.10   0.07
```

THE MONOCHROMATOR AND BACKGROUND CORRECTIONS

Comparison of several PBT pole figures indicates that a background correction must be made to get an accurate pole figure. Best results will be obtained with a continuous scan (IM mode) but /FB will give a satisfactory result in a shorter time. The monochromator gives a cleaner pole figure than one gets with filtered radiation, but its use is not mandatory.

COMBINING TRANSMISSION AND REFLECTION POLE FIGURES WITH POLEDAT

The POLEDAT PROGRAM (Desper, 1978) will combine transmission and reflection pole figures to give a complete pole figure. There are two requirements. One must make the volume correction (and absorption correction if needed), and it is imperative that the angle ranges and increments be compatible for both measurements. Use the same increment for ANGLE 1 and ANGLE 2 for both transmission and reflection.

For transmission (MTHD=1) omega must start at zero. It may increase or decrease. Chi is not restricted but if you want only one quadrant, start at zero and go to 90°.

For reflection (MTHD=0), phi must start at zero and increase. Chi usually starts at 40° and increases to 90°. This gives sufficient overlap with the transmission data to complete the pole figure.

The MERGE feature of POLEDAT has not been tested at Wright-Patterson Air Force Base.

USING POLEDAT

The CONLEV card should be used if CALCOMP contour plots are to be drawn.

The ABTH card must be used to get the volume correction. The input parameter is 0 if no absorption correction is desired. Absorption, μ , can be conveniently measured with the monochromator in place by determining I_0 (no sample) and I with the sample in the direct beam (be sure to restrict the beam to protect the counter). Remember $I/I_0 = e^{-\mu t}$.

Set KOORD=1 for samples with the machine direction mounted parallel to the PHI axis and the Sheet normal parallel to the Chi axis. This is the usual arrangement.

THE ORIENTATION FACTOR

The orientation factor for a pole figure analysis is calculated by POLEDAT, but only if data for a complete pole figure is available. One can get the orientation factor for transmission data if the program is given the extra data. This can be done by editing the data file with the PRIME 850 editor. Change the angle limits and supply the extra data points with zero or very small values. (It goes without saying that the pole density in the region of fabricated data had better be zero or you will be in trouble). POLEDAT thus fooled will give the orientation factors.

For the transmission case with KOORD=1 the average squared direction cosines for the x, y, and z axes will be:

1. If the poles are at $\omega=0$ and $\chi=90$

$$(\cos_x^2 \phi) = 0$$

$$(\cos_y^2 \phi) = 0$$

$$(\cos_z^2 \phi) = 1$$

2. If the poles are at $\omega=0$ and $\chi=0$

$$(\cos_x^2 \phi) = 0$$

$$(\cos_y^2 \phi) = 1$$

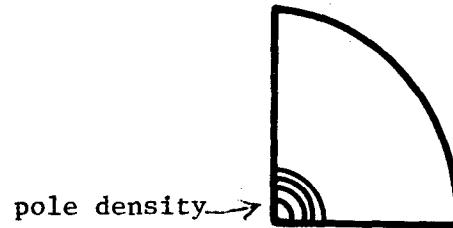
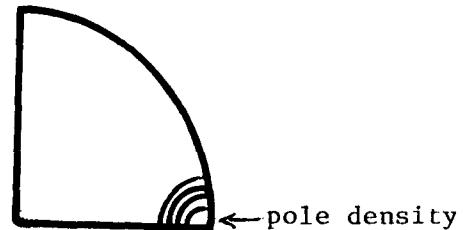
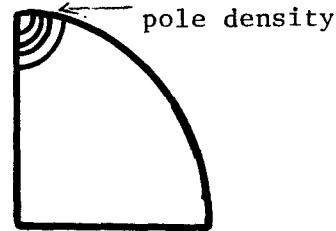
$$(\cos_z^2 \phi) = 0$$

3. If the poles are at $\omega=90$ and $\chi=0$ to 90

$$(\cos_x^2 \phi) = 1$$

$$(\cos_y^2 \phi) = 0$$

$$(\cos_z^2 \phi) = 0$$



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